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IS 14575 (1999): Determination of pentacholorophenol (PCP) in leather - Method of test [CHD 17: Leather, Tanning Materials and Allied Products]

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भारतीय मानक

चमड़े में पेन्टाक्लोरोफेनल (पी सी पी) का निर्धारण — परीक्षण पद्धति

Indian Standard

DETERMINATION OF PENTACHLOROPHENOL (PCP) IN LEATHER — METHOD OF TEST

ICS 59.140.99

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN. 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1999

Price Group 4

Leather Sectional Committee, CHD 017

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather Sectional Committee had been approved by the Chemical Division Council.

Pentachlorophenol (PCP) was being used as preservative in leather as well as in many other leather processing chemicals in the past. However, use of this chemical has now been banned and limits of PCP in leather has been restricted in almost all over the world because of its suspected carcinogenic effect. In India also use of this chemical has been banned by the Government of India through the Gazette Notification number 17-10/91-PPI dated 17 May 1991. In view of this ban detection and determination of PCP has become of utmost importance.

Further it has been observed during the study that even though PCP is not used in the chemicals used for processing of leather but because of usage of PCP over a prolonged period in the past the real life data gives about 5 ppm PCP/kg of leather. It may require some more time to be flushed out from the system completely.

The Leather Sectional Committee of the Bureau therefore decided to formulate a method of test on this subject. So far there has been no universally accepted method for determination of PCP. This method has been developed by the Central Leather Research Institute, Chennai and the PFI, Germany and is known as Indo-German method (Method B). This method is at present under the consideration of ISO for adoption as an International Standard.

In addition to the above method two more methods nave also been incorporated in this standard keeping in view the other international methods being followed all over the world for determination of PCP in leather. However, in case of any dispute, Method A shall be considered as referee method.

Composition of the Committee responsible for formulation of this standard is given in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DETERMINATION OF PENTACHLOROPHENOL (PCP) IN LEATHER — METHOD OF TEST

1 SCOPE

This standard prescribes methods of test for determination of Pentachlorophenol (PCP) in leather. The three methods described in this standard are applicable to all kinds of leather and raw hides.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards indicated below:

IS No.	*Title*
582 : 1970	Methods of chemical testing of leather (*first revision*)
1640 : 1960	Glossary of terms relating to hides, skins and leather
5868 : 1983	Method of sampling for leather (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1640 shall apply.

4 TEST METHODS

In the event of any dispute the method described in **4.1** (Method A) shall be considered as referee method.

Sampling shall be done in accordance with IS 5868. For every leather sample at least two determinations shall be made.

4.1 Method A

4.1.1 *Principle*

PCP from a leather sample to be analysed is successively acidified with sulphuric acid, washed twice combined with water and then extracted with acetone by Soxhlet extraction. Acetone extract is evaporated combined with sulphuric acid extract and re-extracted with *n*-hexane. The *n*-hexane extract is evaporated to dryness and subjected to acetylation with acetic anhydride and triethylamine at 60°C and worked up to get the acetyl derivative. The acetylated PCP is determined quantitatively by Gaschromatograph with electron capture detector (GC-ECD) using Tribromophenol acetate (TBPA) as internal standard.

4.1.2 *Reagents*

All reagents shall be of analytical grade purity.

4.1.2.1 *Sulphuric acid*

4.1.2.2 *Acetone*

4.1.2.3 n-*Hexane*

4.1.2.4 *Acetic anhydride*

4.1.2.5 *Triethyl amine*

4.1.2.6 *Sodium sulphate*

4.1.2.7 *Pentachlorophenol (PCP)*

4.1.2.8 *Tribromophenol acetate (TBPA)*

4.1.3 *Apparatus*

4.1.3.1 *Soxhlet extractor*

4.1.3.2 *Rotavapour*

4.1.3.3 *Water bath* (60°C)

4.1.3.4 *Separating funnels* (60 ml and 120 ml)

4.1.3.5 *Microlitre syringe* (10 μl)

4.1.3.6 Gaschromatograph designed for capillary columns, with electron capture detector (GC-ECD).

4.1.4 *Procedure*

About 5 g of the leather sample pieces are accurately weighed in a beaker to the nearest 0.001 g and soaked with 10 ml of 10 percent sulphuric acid for 1 hour after which the sulphuric acid is decanted in a clean beaker, and the leather pieces are washed two times with 25 ml of distilled water. The sulphuric acid solution washings are combined and kept separately. The leather sample is then extracted with 150 ml acetone by Soxhlet extraction for 6 hours. The acetone extract is then concentrated to a small volume and combined with dilute sulphuric acid extract and water washings. This is then re-extracted 4 times with 25 ml hexane. The hexane extract is dried over sodium sulphate, filtered and evaporated under vacuum nearly to dryness in a rotavapour and the residue is taken for acetylation.

The above residue is treated with 1 ml acetic anhydride and 0.5 ml triethylamine and heated on a water bath at 60°C for 30 min. The resulting acetylated product is taken in 10 ml hexane and 5 ml distilled water in a separating funnel, shaken well and the layers were separated. The aqueous layer is re-extracted with 2 times 10 ml hexane and the combined hexane solution is dried over sodium sulphate and evaporated to about 7 ml in a rotavapour. 1 ml of internal standard containing 50 µg of Tribromophenol acetate is added and the total volume is made up to 10 ml.

The sample is now ready for GC injection.

4.1.5 *Gaschromatographic Conditions (Examplary)*

Capillary Gaschromatograph

Column : stationary phase : fused silica capillary with medium polarity (95 percent methyl and 5 percent phenyl silicone)
Length : 25 m
Inner diameter : 0.2 mm
Film thickness : 0.25 µm

Temperature Conditions

Injector	300°C
Column oven	200°C
Detector	300°C
Detector	Electron capture detector, ECD (Ni-63)
Carrier gas/ make-up gas	nitrogen
Injection volume	1 µl

4.1.6 *Calibration*

A standard solution containing 58 µg of PCP acetate and 50 µg of TBP acetate in 10 ml hexane is prepared by diluting stock solutions. From this 1 µl is injected by means of a microlitre syringe and the chromatogram is recorded under the conditions mentioned above. The retention time of PCP acetate and TBP acetate are recorded. From peak areas of PCP acetate and TBP acetate the area ratio is calculated for the standard sample.

4.1.7 *Results*

From the peaks obtained PCP acetate and TBP acetate are identified by their retention times as well as the relative retention times. Area ratio of the PCP acetate and TBP acetate peak is determined.

PCP content of the sample in mg/kg:

$$C_{PCP} = \frac{50}{A_{PCPA_{Standard}} / A_{TBPA_{Standard}}} \times \frac{A_{PCPA_{Sample}} / A_{TBPA_{Sample}}}{m}$$

where

A = peak area from gaschromatogram,
$PCPA$ = pentachlorophenol acetate,
$TBPA$ = tribromophenol acetate, and
m = mass of sample.

PCP content of leather on moisture-free or dry basis in mg/kg:

$$C_{PCP_{Dry}} = \frac{C_{PCP} \times 100}{100 - W}$$

where

W = moisture content of sample when tested in accordance with LC:2 of IS 582, in percent.

4.2 Method B

4.2.1 *Principle*

The ground leather is extracted with a polar solvent in a Soxhlet apparatus after adding tetrachloroguaiacol (TCG) as internal standard. The extract is cleaned from contaminants which are co-extracted from the sample by a column chromatography clean-up procedure. Neutral, non-hydrolysable compounds which passes the column are separated in a second clean-up step by liquid-extraction. The PCP and other phenols are acetylated with acetic anhydride in a potassium carbonate solution. The acetylated phenols are separated by capillary gaschromatography and are detected by ECD.

4.2.2 *Reagents*

All regents shall be of analytical grade purity.

4.2.2.1 *Acetone*

4.2.2.2 *Sodium hydroxide*

4.2.2.3 *Sodium sulphate anhydrous*

4.2.2.4 *Sulphuric acid (concentrated)*, 95-97 percent purity.

4.2.2.5 *Celite 545 purified and glowed*

4.2.2.6 n-*Hexane*

4.2.2.7 *Acetic anhydride*

4.2.2.8 *Potassium carbonate solution*

4.2.2.9 *Pentachlorophenol PCP*, melting point, 188-190°C.

4.2.2.10 *Tetrachloroguaiacol TCG,* melting point, 118-119°C.

4.2.2.11 *Triethylamine*

4.2.2.12 *Standard solution K*1: 5.00 µg, PCP/ml acetone.

4.2.2.13 *Standard solution K*2, 5.00 µg TCG/ml acetone.

4.2.2.14 *Standard solution K*3, 1.00 µg PCP/ml *n*-hexane.

4.2.2.15 *Standard solution K*4, 1.00 µg TCG/ml *n*-hexane (*see* Note).

4.2.2.16 *Hydrogen, purity,* 99.99 percent.

4.2.2.17 *Argon/methane gas mixture* 95:5, purity 99.99 percent.

NOTE — The stability of the standard solutions is limited to 8 weeks, because of the tendency of the solutions to precipitate the solved compounds, the solutions shall not be stored in a refrigerator.

4.2.3 *Apparatus*

4.2.3.1 *Soxhlet apparatus*

4.2.3.2 Chromatographic column with teflon stopcock and fritte, diameter 1-2 cm, length 20-30 cm.

4.2.3.3 Temperature programmable gaschromatograph for capillary columns with Electron Capture Detector (ECD) preferable with automatic sampler.

4.2.4 *Procedure*

4.2.4.1 *Extraction*

Accurately weigh out 5 g ground leather to the nearest 0.001 g. Pack it into an extraction thimble placed inside the Soxhlet apparatus. Give 5 ml of solution *K*2 onto the leather and add 200 ml acetone and one anti-bumping granule.

Extract for 50 ± 5 solvent changes. The extraction time shall not exceed 16 h. The extract is quantitatively transferred to a 250-ml volumetric flask and made up to volume with acetone. 5 ml of this extract is taken and evaporated down to about 1 ml using a rotary evaporator. The residue is acidified with one drop of sulphuric acid. Add 5 ml *n*-hexane and evaporate again. This procedure has to be repeated until the extract is acetone-free.

4.2.4.2 *Column chromatographic clean-up*

The final volume of the acetone-free extract in *n*-hexane should be about 5 ml. This is the solution used for the clean-up procedure on the celite-sulphuric acid column.

Preparation of celite-sulphuric acid column:

80 g glowed, purified and conditioned (analytical grade) celite 545 are mixed intensively with 60 ml of sulphuric acid (preferably in a mortar). The mixture is highly hygroscopic and shall be used just after preparation.

The chromatographic column is immediately filled with celite-sulphuric acid mixture up to 17-18 cm and filled up with *n*-hexane. The surface is lightly compressed with a glass rod. Add some glass beads or glass wool to avoid suspension at the column head. The column is rinsed with 80 ml *n*-hexane. The *n*-hexane sample solution is transferred; quantitatively to the column. Elute with 150 ml *n*-hexane with a flow rate of (2 ± 0.5) ml/min. Transfer the eluate into a separating funnel.

4.2.4.3 *Liquid-liquid extraction*

The eluate in the separating funnel is extracted two times with 20 ml of potassium carbonate solution (preferably on a suitable machine for 5 minutes each). Allow to separate each time and discard the *n*-hexane layer. The separated aqueous layers were combined in a 100-ml separating funnel.

4.2.4.4 *Derivatisation*

Add 30 ml *n*-hexane and 1 ml acetic anhydride and 0.5 ml triethylamine. Carefully shake under releasing of the pressure. Afterwards shake vigorously on a suitable machine for 30 ± 5 minutes. Allow to separate (normally not longer than 2 minutes). The aqueous layer is separated and washed twice with *n*-hexane. Then the organic layers are combined, dried over sodium sulphate, and filtered to a 100-ml round bottom volumetric flask and made up to volume with *n*-hexane. This is the solution for the gaschromatographic determination.

4.2.5 *Gaschromatographic Procedure*

A gaschromatograph with ECD, as commonly used for capillary-GC is applied.

An example of operational parameters are:

Injection volume	: 1 µl
Temperature programme	: 80°C for 5 minutes up to 250°C with 6°C/minute, 10 minutes at 250°C
Separation column	: fused silica capillary with medium polarity 95 percent Dimethyl — 5 percent diphenylpolysiloxane.
Length	: 30 m
Inner diameter	: 0.32 mm
Film thickness	: 0.25 µm
Carrier gas	: Hydrogen
Make up gas	: Argon/Methane 95:5
Injector temperature	: 250°C
Injector method	: split
Detector	: Electron Capture Detector ECD, Ni-63
Detector temperature	: 270°C

The gaschromatographic conditions shall be as such that a minimum resolution of 5 for PCP and TCG-acetate is obtained.

4.2.6 *Calibration*

4.2.6.1 *Preparation of the calibration solutions*

5 g of PCP-free leather or 5 g of a leather substitute (Highly cross linked vinylimidazol/vinylpyrolidon copolymer 'DIVERGAN') is put into a thimble of a Soxhlet apparatus. 5.00 ml standard solution $K1$ and 5.00 ml $K2$ is added. Then run the complete procedure as described above for the sample.

4.2.6.2 *Calibration of the apparatus*

To calibrate the GC-ECD for the analysis of phenol derivatives establish gaschromatographic operating conditions equivalent to those established for the analysis of the sample solutions. Prepare calibration standards at a minimum of 5 concentration levels by adding volumes of stock standards to a volumetric flask and diluting the volume with *n*-hexane.

The amount of PCP-acetate, TCG-acetate standards may be, for example, between 0.1 mg and 50 mg/10 ml.

NOTE — The described method is developed for a range of 1 to 50 mg/kg PCP in leather and 1 µl injection volume and a capillary column with ECD. The method is also suitable for higher concentrations if the solution obtained from the leather samples are diluted, or smaller aliquots from the leather extracts are taken. It is important to operate within the linear range of the ECD.

4.2.7 *Test of the Analytical System*

4.2.7.1 *Purity of the used chemicals and apparatus*

Care should be taken that the signal of a compound to be measured is not interfered or superimposed by signals of other compounds or contaminants.

All used chemicals with the exception of the standard solutions $K1$, $K2$, $K3$, $K4$ are treated in the same way as the sample itself. If in case of blank runs of the procedure (that means extraction, clean-up procedure a gaschromatographic determination without a leather sample) signals are found at the expected retention times of PCP acetate and TCG acetate it is necessary to take different and purified chemicals (or glassware) or to choose a different separation column which is able to separate the interfering compounds from the compounds to be measured.

4.2.7.2 *Recovery rate after liquid chromatographic clean-up procedure*

1 000 µl of the standard solution $K3$ and $K4$ are added to 150 ml *n*-hexane in a 250-ml separating funnel. This solution is treated in the same way than the sample beginning with **4.2.4.3**.

The recovery rate of TCG and PCP of the solutions from **4.2.6.1** shall be minimum 80 percent of the solution.

4.2.8 *Performance Characteristics of the Procedure*

The following details have been obtained from an inter-laboratory trial:

Concentration level: 3 mg/kg
r = 1.00 mg/kg δ_r : + 0.35 mg/kg
R = 1.17 mg/kg δ_R : + 0.41 mg/kg

Concentration level: 7 mg/kg
r = 1.92 mg/kg δ_r : + 0.68 mg/kg
R = 2.35 mg/kg δ_R : + 0.83 mg/kg

r = repeatability
R = reproducibility

4.2.9 *Calculation and Expression of Results*

PCP-content of leather is determined by using internal and external standard calibration factors. The result is given as PCP content in leather in mg/kg.

$$C_{\mathrm{PCP}} = \frac{A_{\mathrm{PCP}} \times C_{\mathrm{K1}} \times V \times F_{\mathrm{TCG}}}{A_{\mathrm{K1}} \times E}$$

where

A = peak area;
C = concentration in µg/ml;
E = mass of sample,;
V = volume in ml to which the sample was made up (end volume); and
F_{TCG} = calibration factor,

$$F_{\mathrm{TCG}} = \frac{A_{\mathrm{K2}_{\text{solution for calibration}}}}{A_{\mathrm{K2}_{\text{sample solution}}}}$$

Indices:

$K1$ = PCP Standard
$K2$ = TCG Standard

Content of PCP based on dry matter in mg/kg:

$$C_{\mathrm{PCP}_{\mathrm{dry}}} = C_{\mathrm{PCP}} \times F$$

where

$$F = \frac{100}{100 - W}$$

where

W = moisture content of sample when tested in accordance with LC:2 of IS 582, in percent.

4.3 Method C

4.3.1 *Principle*

PCP is extracted from the ground leather sample after adding Tetrachloroguaiacol (TCG) as internal standard and converted to its sodium salt *in situ* using a Soxhlet apparatus containing ethanol and sodium hydroxide.

After evaporating the Soxhlet extract to 5-10 ml, the concentrate is taken up in sodium hydroxide solution. Fats and other neutral materials are then extracted into ether and discarded.

Following this, the aqueous extract is brought to *p*H 1-2 using hydrochloric acid, thereby liberating the free phenol this is subsequently extracted into ether, leaving any ionic contaminants such as emulsifiers in the aqueous phase.

Finally, the ether extract is evaporated to dryness and treated, with Iodomethane, thereby converting phenols to their ethoxy derivative. The sample solution is then analysed by gaschromatography, utilising mass selective.

4.3.2 *Reagents*

4.3.2.1 *Pentachloroanisole* (pentachloromethoxy-benzene), 99.3 percent purity.

4.3.2.2 *Tetrachloroguaiacol* (*1*-methoxy-tetrachlorophenol)

4.3.2.3 *Iodomethane*, stabilized with copper.

4.3.2.4 *Hexachlorobenzene* (HCB)

4.3.2.5 *Anhydrous potassium carbonate*

4.3.2.6 *Sodium hydroxide pellets*

4.3.2.7 *Sodium hydroxide solution*, 0.1 molar.

4.3.2.8 *Hydrochloric acid*, 2.0 molar.

4.3.2.9 *TCG marker solution*

25.0 mg TCG dissolved in 500 ml methanol.

4.3.2.10 *HCB internal standard*

50.0 mg HCB dissolved in 500 ml hexane.

4.3.2.11 *Calibration standards*

25.0 mg pentachloroanisole dissolved in 250 ml hexane.

4.3.3 *Apparatus*

4.3.3.1 *Capillary gaschromatograph with mass selective detector*

4.3.3.2 Pressure tube heavy walled borosilicate glass with teflon screw-in plug, outer diameter 25.4 mm, 35 ml volume, 17.8 cm long.

4.3.3.3 Soxhlet extraction apparatus with cellulose extraction thimbles.

4.3.4 *Procedure*

4.3.4.1 *Sample extraction and clean-up*

a) Accurately weigh out 5 g of finely ground leather sample. Loosely pack into an extraction thimble and place inside Soxhlet apparatus.

b) To the Soxhlet flask, add 200-ml methanol, 4 pellets (0.4-0.5 g) of sodium hydroxide and 2.0 ml of TCG marker solution.

c) Soxhlet extract for 80 solvent changes (approximately 8 hours).

d) Evaporate the Soxhlet extract down to a volume of approximately 5 ml using a rotary evaporator or steam bath. Beware of bumping.

e) Add 10 ml of 0.1 M sodium hydroxide solution to the flask. Warm with a swirling motion over a steam bath to re-dissolve any deposited solids (possibly seen as 'tide-marks'). Transfer the alkaline solution into a 250-ml separating funnel.

f) Add another 10 ml of 0.1 M sodium hydroxide solution to the empty flask, warm with a swirling motion, and add to the contents of the separating funnel.

g) Repeat stage (f).

h) Stopper and shake separating funnel vigorously for one minute. Measure *p*H. If it is outside the range 12-13, adjust accordingly with a further aliquot of alkali.

j) Add 40 ml diethyl ether to contents of separating funnel. Stopper and shake gently by hand for 5 minutes, releasing the pressure at regular intervals by slowly opening up the tap.

k) Suspend separating funnel in a ring clamp and allow 10 minutes for separation of the phases.

m) Run off bottom layer (including boundary layer) into the original Soxhlet flask. Discard the remaining ether phase.

n) Transfer contents of Soxhlet flask back into the emptied separating funnel, and repeat stages (j) and (k).

p) Run the bottom (aqueous) phase into Schott bottle, ensuring that the boundary phase remains in the separating funnel with the ether phase. Now discard the contents of the separating funnel.

q) Pipette 10 ml of 2 M hydrochloric acid into the Schott bottle containing the aqueous phase, shake for 30 seconds, and measure the *p*H, which should be in the range 1-2. If necessary, adjust to within this range using a further, aliquot of acid.

r) Stopper the container, and shake vigorously on an autoshaker for 20 minutes.

s) After the period of shaking, ensure that the *p*H has remained within the range 1-2.

t) Add 40 ml of ether to the contents of the Schott flask, and agitate at medium, speed on an auto-shaker for 20 minutes.

u) Allow to stand for 10 minutes for separation of the phases.

v) Using an auto-pipette, transfer the top layer (ethereal solution of the analytes) into a clean round bottomed flask.

w) Carry out stages (t) to (v) (both inclusive) twice more, combining the three ether extracts in the same round bottomed flask.

x) Gently evaporate the ether in the flask to approximately 5 ml using a steam bath in a fume cupboard.

y) Evaporate off all remaining ether using a steam of dry nitrogen.

z) Proceed with the sample derivatisation, as detailed in section **4.3.4.3**.

4.3.4.2 *Procedure for emulsion forming extracts*

Continued from step k, of **4.3.4.1**:

m) Quantitatively transfer the contents of the separating funnel into a Schott screw top bottle or equivalent, and secure the top.

n) Centrifuge at 2 500 rpm for 30 min.

p) Using an auto-pipette or other suitable device, remove and discard the entire upper layer.

q) Pierce auto-pipette tip through the pad-like boundary phase and quantitatively transfer the entire aqueous phase to a clean Schott bottle or equivalent.

r) Agitate the flask briskly on an auto shaker for 5 min, and then allow 10 min for the phases to separate. Centrifugation may or may not be required at this stage.

s) Discard upper (ether) phase, as for stage (p) of this section and combine bottom layer with aqueous phase from (q) above.

t) Proceed from stage (q) in **4.3.4.1**.

4.3.4.3 *Sample derivatisation*

a) Using 10-15 ml of acetone, the residue obtained from the final stage of the clean-up procedure is washed quantitatively into the oven-dried pressure tube containing 2 g of anhydrous potassium carbonate.

b) Pipette 5 ml of Iodomethane into the pressure tube and tightly screw down the top.

c) Shake the pressure tube vigorously for 30 seconds.

d) Heat the sealed pressure tube over the steam bath for a period of 20 minutes.

e) Shake the pressure tube and cool under a cold running tap.

f) Quantitatively transfer the entire contents of the tube into a round bottomed flask using acetone.

g) Pipette in 1.0 ml of HCB internal standard solution.

h) Evaporate off the bulk of acetone over the steam-bath, taking nearly to dryness.

j) Wash the remainder through a filter into a 10- ml volumetric flask using 2 ml aliquots of hexane. Make up to volume with hexane.

k) The sample is now ready for analysis by gaschromatography, utilising mass selective detection (mass spectroscopy).

4.3.4.4 *Control*

a) Pipette 2.0 ml of TCG marker solution into the dry pressure tube, and evaporate to dryness with a stream of nitrogen.

b) Add 2 g of anhydrous potassium carbonate followed by 10-15 ml of acetone.

c) Proceed from stage (b) of **4.3.4.3**.

The control sample is now analysed by gas-chromatography together with the sample(s), and the full range of standards.

4.3.5 *Gaschromatographic Conditions*

Capillary gaschromatograph with mass selective detector

Column : 25 m, 0.22 mm ID, BP5 (cross-linked 5 percent phenyl methyl silicone gu.), with film thickness 0.2-5 Am

Carrier gas : Helium, flow rate 0.76 ml/min (linear velocity 33.5 cm/sec) at a column head pressure of 0.07 kg/cm^2

Injector temperature : 240°C

Injection mode : Splitless

Splitless time : 2.0 minutes

Injection volume : 0.5 µl

Temperature programme : 2 minutes at 50°C, 20°C/min to 110°C, then 2°C/min to 150°C, with finally 25°C/min to 250°C (Programme time 29 minutes)

Analyser Temperatures

Transfer line (interface)
Temperature : 280°C

Ion source temperature : 190°C (approximately)

Quadruple temperature : 140°C (approximately)

Electron multiplier temperature : 65°C (approximately)

4.3.6 *Results*

Concentration of PCP in the leather sample, mg/kg:

$$C_{PCP} = (C_{PCP_{extract}}) \times \frac{V_{extract}}{W} \times \frac{(A_{TCG_{Control}} / A_{HCB_{Control}})}{(A_{TCG_{extract}} / A_{HCB_{extract}})}$$

where

A = peak area,

V = extraction volume in ml,

m = weight of leather sample in g, and

$C_{PCP_{extract}}$ = obtained from calibration graph in mg/ml.

Content of PCP based on dry matter in mg/kg:

$$C_{PCP_{dry}} = C_{PCP} \times F$$

where

$$F = \frac{100}{100 - W}$$

where

W = moisture content of sample when tested in accordance with LC:2 of IS 582, in percent.

4.4 Test Report

The report shall include:

a) identification details of the leather,

b) reference to this Indian Standard and to the method used (refer to this standard and Methods A, B or C),

c) the result in mg/kg to one decimal place of at least two determinations and the mean value,

d) details about any deviations from the described procedure, and

e) moisture content of the leather (*see* IS 582).

NOTES

1 The result in percent by mass is based on dry matter especially for wet leathers, for example, Wet Blues.

2 The water content of the leather is determined from a sample of leather which has been taken just beside the sample for the PCP determination. The samples which has been used for the determination of the moisture content shall not be used for the determination of the PCP content. Wet leathers are dried in a standard atmosphere before the determination of the moisture content according to IS 582. The loss of weight in standard atmosphere and after drying according to IS 582 are added.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Leather Sectional Committee, CHD 017

Chairman	*Representing*
Dr T. Ramasami	Central Leather Research Institute, Chennai
Members	
Dr S. Sadulla (*Alternate* to Dr T. Ramasami)	
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